



UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
WASHINGTON, D.C. 20460

OFFICE OF
PREVENTION, PESTICIDES, AND
TOXIC SUBSTANCES

PC Code: 118203

DP Barcode: 349859, 356464, 364059

MEMORANDUM

June 30, 2009

SUBJECT: **Saflufenacil:** Transmittal of Data Evaluation Records for Environmental Chemistry Methods.

TO: Kathryn Montague, Product Manager
HB/RD (7505P)

FROM: Greg Orrick, Environmental Scientist
ERB4/EFED (7507P)

Greg Orrick 6-30-09

THROUGH: Elizabeth Behl, Chief
ERB4/EFED (7507P)

M. Echeverria (for E. Behl) 6-30-09

This memo is to inform you that two (2) DERs for saflufenacil are finalized. Two electronic files are associated with these finalized DERs. Study MRIDs and classifications are listed in **Table 1** below.

Table 1. DERs and Classifications for Environmental Chemistry Methods for Saflufenacil.

MRID	Study Type	DER Electronic File Name	Study Classification
47127928	Analytical method in water	(No DER: missing ILV)	Upgradeable
47127831	Analytical method in soil	(No DER: replaced by MRID 47699902)	Upgradeable
47523802	Analytical method in water	(No DER: replaced by MRID 47699903)	Upgradeable
47699902 47127832	Analytical method and ILV in soil	118203 47699902+ ECM - Soil.pdf	Supplemental
47699903 47523803	Analytical method and ILV in water	118203 47699903+ ECM - Water.pdf	Acceptable

BAS 800 H in Soil/2008-0431/118203/BASF Corporation/241

PMRA Submission Number: 2008-0431/Company Code: BAZ

Active Code: SFF/Use Site: 13 and 14

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(Revision 2)

Data Requirement: PMRA Data Code: 8.2.2.1
PMRA Document No.:
EPA DP Barcode: 350054
OECD Data Point: IIA.4.4.
EPA Guideline: ECM Method Review

Test material:

Common name: Saflufenacil (BAS 800 H)
CAS name: 2-chloro-5-[3,6-dihydro-3-methyl-2,6-dioxo-4-(trifluoromethyl)-1(2H)-pyrimidinyl]-4-fluoro-N-[[methyl(1-methylethyl)amino]sulfonyl]-benzamide
IUPAC name: N'-[2-chloro-4-fluoro-5-[1,2,3,6-tetrahydro-3-methyl-2,6-dioxo-4-(trifluoromethyl)pyrimidin-1-yl]benzoyl]-N-isopropyl-N-methylsulfamide

EPA Primary Evaluator: Elizabeth Flynt Date: 6/30/09
Elizabeth Flynt, Chemist

EPA Peer Reviewer: Charles Kennedy Date: 6/30/2009
Charles Kennedy, Chemist

QA Officer: Christian Byrne Date: 6/30/09
Dr. Christian Byrne, QA Officer

PMRA Global Reviewer: Katherine Keppel-Jones Date: 6/30/09
Katherine Keppel-Jones, HC-PMRA-CES

APVMA Global Reviewer: D. Murphy Date: 6/30/09
Dr. Daryl Murphy, DEWHA/APVMA

ANALYTICAL METHOD: MRID No. 476999-02 / PMRA No.1731026, Saha, M., March 16, 2009, "Method Validation of BASF Analytical Method D0503 entitled: The Determination of Residues of BAS 800H and Its Metabolites, in Soil Using LC/MS/MS",

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Metabolites, in Soil Using LC/MS/MS (MRID No. 471278-32 / PMRA No. 15469-45)
was conducted by Adpen Laboratories at Jacksonville, FL. Pages 1-203. The BASF
Study No. is 132662.

EXECUTIVE SUMMARY

The method is applicable for the quantitative determination of residues of BAS 800 H and its metabolites in soil. The method was created in accordance with EPA's Good Laboratory Practice Standards, Title 40 Code of Federal Regulations Part 160. Although, the ECB found that this Environmental Chemistry Method (ECM) and its associated independent laboratory validation (ILV) met all criteria for a scientifically valid method, it is considered supplemental because the LOQ validated (0.01 mg/kg) is significantly higher than the lowest phytotoxic endpoint in soil (0.0005 mg/kg).

Method Summary

Soil samples are extracted by vortexing with acetonitrile twice, and then acetonitrile:water twice, followed by centrifugation, and removal of the supernatant for analysis. The residues are determined using LC/MS/MS.

The reported limit of quantification was found to be 0.01 mg/kg for all analytes.

METHOD ACCEPTABILITY/DEFICIENCIES/CLARIFICATIONS

Although the method was well documented and meets all the requirements for a scientifically valid method there are several inconsistencies/corrections which should be noted.

The LOQ used in this validation was 0.01 mg/kg (ppm), whereas in order to analyze for the lowest phytotoxicity endpoint in soil, the analytical method would need an LOQ of approximately 0.0005 mg/kg (0.0005 ppm). Therefore, although the method is valid, it is considered supplemental.

On page 101 of the amended registrant method, the LOQ is correctly stated as 0.01 ppm, but the example calculation, "Therefore, at the LOQ, if the amount of analyte is 10 pg on column, the LOD is 2 pg on column." uses an arbitrary final value that is different than the value in the method. The reviewers find this unnecessarily confusing.

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COMPLIANCE

Signed and dated statements that this method was conducted in accordance with the requirements for Good Laboratory Practice Standards, 40 CFR 160 were present in the method. A statement of non-confidentiality on the basis of the method falling within the scope of FIFRA Section 10 (d)(1)(A), (B), or (C) was present.

A. BACKGROUND INFORMATION

BAS 800 H is a herbicide that will be used for the treatment of cotton, cereal and other crops in the U.S.

TABLE A.1. Test Compound Nomenclature for Saflufenacil	
Compound	Chemical Structure *See Appendix A for the chemical structure information
Common name	Saflufenacil (BAS 800 H)
Company experimental name	BAS 800 H
IUPAC name	N'-[2-chloro-4-fluoro-5-[1,2,3,6-tetrahydro-3-methyl-2, 6-dioxo-4-(trifluoromethyl)pyrimidin-1-yl]benzoyl]-N-isopropyl-N-methylsulfamide
CAS Name	2-chloro-5-[3,6-dihydro-3-methyl-2,6-dioxo-4-(trifluoromethyl)-1(2H)-pyrimidinyl]-4-fluoro-N-[[methyl(1-methylethyl)amino]sulfonyl]-benzamide
CAS #	372137-35-4

TABLE A.2. Physicochemical Properties of the Technical Grade Test Compound	
Parameter	Value
Melting point/range	Not available
pH	Not available
Density	Not available
Water solubility (25 °C)	Not available
Solvent solubility (mg/ml at 20 °C)	Not available
Vapour pressure	Not available
Dissociation constant (pK _a)	Not available
Octanol/water partition coefficient	Not available
UV/visible absorption spectrum	Not available

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B.1. Principle of Method

A 0.1 gram aliquot of soil sample is placed into a well plate tube and fortified with an appropriate volume of spiking solution. A volume of 0.4 ml of acetonitrile is added and the well tube capped and vortexed twice (once up side down, then right side up) at 2400 rpm for 1 minute each. Then an additional 0.4 ml of acetonitrile:water (40:60 v/v) is added and the well tube capped and vortexed twice (once up side down, then right side up) at 2400 rpm for 1 minute each. The vortexing is followed by centrifugation at 3000 rpm for 5 minutes. The supernatant is removed and transferred into a Matrix Alpha Numeric Tube. Sample and controls are analyzed via LC/MS/MS.

TABLE B.1.1.	Summary Parameters for the Analytical Method Used for the Quantitation of Chemical Residues in Matrices Studied
Method ID	ECM0242S1-S7
Analyte(s)	BAS 800 H, M800H01, M800H02, M800H07, M800H08, M800H15, M800H22
Extraction solvent/technique	Soil samples are extracted using acetonitrile and acetonitrile:water
Cleanup strategies	Centrifugation
Instrument/Detector	PE Series 200 Micro Pump System with Series 200 Autosampler / PE Sciex API 3000 Biomolecular Mass Analyzer

C. RESULTS AND DISCUSSION

C.1.Recovery Results Summary

TABLE C.1.1. Recovery Results from Method Validation of Soil			
Matrix	Spiking Level (conc. units)	Mean% Recoveries	Relative Standard Deviation
*See Appendix B			

C.1.2. Method Characteristics

TABLE C.1.2. Method Characteristics	
Analyte	BAS 800 H, M800H01, M800H02, M800H07, M800H08, M800H15, M800H22
Limit of Quantitation	0.01 ppm
Limit of Detection (LOD)	0.002 ppm
Accuracy/Precision at LOQ	*See Appendix B
Reliability of the Method/ [ILV]	An ILV was performed for this method. MRID No. 471278-32
Linearity	Linear curves were prepared for each analyte. The correlation coefficient was > 0.9940 for all compounds.

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TABLE C.1.2. Method Characteristics	
Specificity	The method is very specific due to the use of MS-MS which is the most highly specific method for detection of residues at low concentration.

C.2. Independent Laboratory Validation (ILV)

TABLE C.2.1. Recovery Results Obtained by an Independent Laboratory Validation of the Method for the Determination of Saflufenacil and its Metabolites in Water		
Matrix	Spiking Level (conc. units)	Recoveries
See Appendix C		

D. CONCLUSION

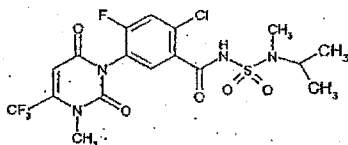
Although this is a well documented method which was confirmed by an independent laboratory validation, ECB finds the method supplemental, because the validated LOQ (0.01 mg/kg) is significantly higher than the lowest phytotoxic endpoint in soil (0.0005 mg/kg).

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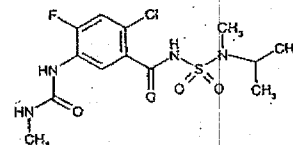
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Appendix A: Chemical Structures of BAS 800 H and Its Metabolites

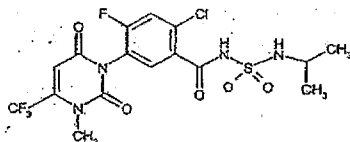
BASF Code Name: BAS 800 H
BASF Registry Number: 4054449
CAS Number: 372137-35-4
Molecular Formula: $C_{17}H_{19}ClF_4N_4O_5S$
Molecular Weight: 500.9
Lot No.: L67-140
Purity: 99.9%
Expiration date: July 01, 2008
Structural Formula:



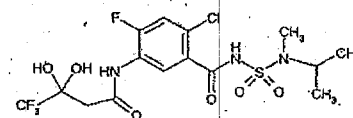
BASF Code Name: M800H07
BASF Registry Number: 4775453
Molecular Formula: $C_{15}H_{16}ClF_4N_4O_5S$
Molecular Weight: 380.8
Lot No.: L67-196
Purity: 95.4%
Expiration date: March 1, 2009
Structural Formula:



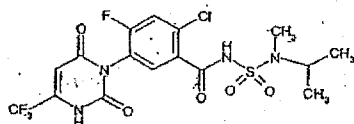
BASF Code Name: M800H01
BASF Registry Number: 4118561
Molecular Formula: $C_{16}H_{15}ClF_4N_4O_5S$
Molecular Weight: 486.8
Lot No.: L74-62
Purity: 98.8%
Expiration date: February 1, 2008
Structural Formula:



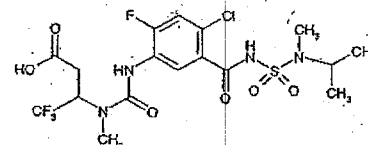
BASF Code Name: M800H015
BASF Registry Number: 5264357
Molecular Formula: $C_{16}H_{16}ClF_4N_5O_5S$
Molecular Weight: 479.9
Lot No.: L74-80
Purity: 94.5%
Expiration date: June 1, 2008
Structural Formula:



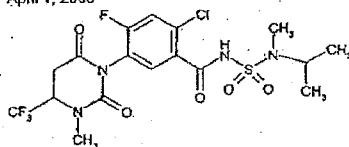
BASF Code Name: M800H32
BASF Registry Number: 4118416
Molecular Formula: $C_{16}H_{15}ClF_4N_4O_5S$
Molecular Weight: 486.8
Lot No.: L67-186
Purity: 99.2%
Expiration date: March 1, 2009
Structural Formula:



BASF Code Name: M800H022
BASF Registry Number: 5216337
Molecular Formula: $C_{17}H_{21}ClF_4N_4O_5S$
Molecular Weight: 520.9
Lot No.: L74-56
Purity: 94.1%
Expiration date: March 1, 2008
Structural Formula:



BASF Code Name: M800H08
BASF Registry Number: 4773881
Molecular Formula: $C_{17}H_{19}ClF_4N_4O_5S$
Molecular Weight: 502.9
Lot No.: L74-66
Purity: 97.2%
Expiration date: April 1, 2008
Structural Formula:



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Appendix B: Summary of Percent Recoveries, Average and RSDs for BAS 800 H and Its Metabolites

Summary of Percent Recoveries, Average and Standard Deviation for Each Analyte in Different Types of Soil (Method D0503) Obtained from Primary Quantitation Ion

Soil Type/ Soil Depth/ Location	Analytes	Fertilization Level (ppm)	Recovery (%)	Average Recovery (%)	Standard Deviation	RSD ¹ (%)
Loamy Sand/0-1" (WA)	BAS 800 H	0.01	101,111.38 110,154	101	7.3	6.8
		0.1	112,87,115 96,109	106	8.7	8.2
		Overall (N=2)		107	7.6	7.1
	M000H01	0.01	84,83.55 92.83	89	5.8	6.4
		0.1	96,82.50 97.95	94	2.9	3.1
		Overall (N=2)		92	5.0	5.4
	M000H02	0.01	90,115,101 112,102	104	9.8	9.4
		0.1	103,96,094 103,102	98	11.3	11.5
		Overall (N=2)		101	10.5	10.3
	M000H07	0.01	92,102.99 89.55	95	5.5	5.8
		0.1	107,95.81 87,102	98	6.2	6.3
		Overall (N=2)		97	5.8	6.0
	M000H06	0.01	97,94,102 110,125	106	12.3	11.6
		0.1	101,112.84 75,102	96	14.4	15.0
		Overall (N=2)		101	13.6	13.5
	M000H15	0.01	115,90.91 115,105	103	12.5	12.2
		0.1	118,94,108 99,96	104	9.3	9.0
		Overall (N=2)		104	10.4	10.1
	M000H22	0.01	112,107,106 101,113	108	5.2	4.8
		0.1	89,92,101 95,104	97	6.1	6.3
		Overall (N=2)		102	7.4	7.5

¹Relative Standard Deviation (RSD) = (Standard Deviation/Average Recovery) x 100

Summary of Percent Recoveries, Average and Standard Deviation for Each Analyte in Different Types of Soil (Method D0503) Obtained from Primary Quantitation Ion

Soil Type/ Soil Depth/ Location	Analytes	Fertilization Level (ppm)	Recovery (%)	Average Recovery (%)	Standard Deviation	RSD ¹ (%)
Sandy Loam/0-6" (Genus 2.2)	BAS 800 H	0.01	79,83.89 91,92	89	5.8	6.6
		0.1	91,102,110 92,111	105	5.4	5.1
		Overall (N=2)		95	10.9	19.4
	M000H01	0.01	73,84.93 83.81	87	5.8	6.7
		0.1	85,83.95 83,103	95	5.3	5.6
		Overall (N=2)		91	5.7	7.4
	M000H02	0.01	82,85,100 83,102	90	7.5	7.2
		0.1	90,103,98 91,94	97	4.8	4.7
		Overall (N=2)		93	6.1	6.7
	M000H07	0.01	75,83,79 95,82	97	5.2	5.5
		0.1	102,96,100 100,93	104	4.6	4.4
		Overall (N=2)		100	6.1	6.1
	M000H06	0.01	103,100 100,95	75	14.5	29.1
		0.1	102,92,105 101,97	101	5.8	5.8
		Overall (N=2)		88	17.5	39.8
	M000H15	0.01	91,86.83 87,81	89	9.9	11.1
		0.1	84,93,94 97,99	89	5.8	6.2
		Overall (N=2)		85	8.5	10.1
	M000H22	0.01	85,114,119 97,97	100	11.4	11.4
		0.1	93,98,97 94,95	93	3.8	3.7
		Overall (N=2)		97	9.9	11.2

¹Relative Standard Deviation (RSD) = (Standard Deviation/Average Recovery) x 100

Summary of Percent Recoveries, Average and Standard Deviation for Each Analyte in Different Types of Soil (Method D0503) Obtained from Primary Quantitation Ion

Soil Type/ Soil Depth/ Location	Analytes	Fertilization Level (ppm)	Recovery (%)	Average Recovery (%)	Standard Deviation	RSD ¹ (%)
Loam/0-1" (CA)	BAS 800 H	0.01	91,102,112,100,95 110,110,106,105,102	104	7.0	6.7
		0.1	93,104,106,103,107 96,97,109,100,117	103	6.8	6.5
		Overall (N=2)		103	6.7	6.5
	M000H01	0.01	112,91,90,85,90 96,94,85,82	95	7.6	8.1
		0.1	100,106,100,100,101 91,83,100,91,98	98	5.1	5.2
		Overall (N=2)		97	6.6	6.8
	M000H02	0.01	109,95,109,105,97 100,107,108,95,100	102	5.7	5.6
		0.1	97,94,109,101,112 89,90,97,98,93	98	7.6	8.0
		Overall (N=2)		100	7.0	7.0
	M000H07	0.01	94,92,100,92,97 97,103,101,104,100	98	5.4	5.5
		0.1	107,102,97,107,107 92,103,95,105,103	102	5.5	5.4
		Overall (N=2)		100	5.5	5.4
	M000H06	0.01	99,95,119,73,99 102,95,112,68,102	98	12.1	12.4
		0.1	100,88,119,77,104 95,75,92,115,111	99	15.7	15.8
		Overall (N=2)		99	13.9	13.8
	M000H15	0.01	96,87,93,90,105 112,93,74,72	90	13.5	15.1
		0.1	105,100,105,11,95 78,105,115,11,123	99	11.7	11.7
		Overall (N=2)		95	13.3	14.1
	M000H22	0.01	92,97,85,84,97,91 109,99,108,100	95	4.7	5.1
		0.1	85,83,90,114,93,90 101,101,99,99	95	9.1	9.6
		Overall (N=2)		95	8.7	9.1

¹Relative Standard Deviation (RSD) = (Standard Deviation/Average Recovery) x 100

Summary of Percent Recoveries, Average and Standard Deviation for Each Analyte in Different Types of Soil (Method D0503) Obtained from Primary Quantitation Ion

Soil Type/ Soil Depth/ Location	Analytes	Fertilization Level (ppm)	Recovery (%)	Average Recovery (%)	Standard Deviation	RSD ¹ (%)
Clay Loam/ 24-39" (CA)	BAS 800 H	0.01	101,102,100 109,100	103	5.3	5.2
		0.1	100,105,96 102,98	101	3.7	3.7
		Overall (N=2)		102	4.4	4.3
	M000H01	0.01	104,94,98 118,94	100	5.1	5.0
		0.1	89,91,102 95,112	98	9.4	9.6
		Overall (N=2)		99	8.8	8.8
	M000H02	0.01	92,89,105 109,87	95	10.6	11.1
		0.1	84,95,96 93,91	91	5.9	6.4
		Overall (N=2)		93	8.2	8.7
	M000H07	0.01	102,105,102 110,91	98	6.0	6.1
		0.1	94,94,95 93,100	96	3.8	3.1
		Overall (N=2)		97	4.7	4.6
	M000H06	0.01	123,100,90 85,92	101	14.5	14.6
		0.1	111,100,93 97,107	95	14.3	15.1
		Overall (N=2)		98	14.0	14.3
	M000H15	0.01	94,88,79 95,86	89	6.9	7.8
		0.1	82,93,96 87,105	92	10.6	11.3
		Overall (N=2)		91	8.5	9.5
	M000H22	0.01	99,99,95 92,94	92	6.4	9.2
		0.1	86,88,96 95,98	96	3.0	3.4
		Overall (N=2)		89	6.5	7.4

¹Relative Standard Deviation (RSD) = (Standard Deviation/Average Recovery) x 100

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Appendix C – Summary of ILV Percent Recoveries and RSDs for BAS 800H and Its Metabolites

Analytes	Fortification Level (ppm)	Average Recovery (%)	RSD
BAS 800 H	0.01	117	7.8
	0.1	102	6.1
M800H01	0.01	108	14.1
	0.1	102	8.3
M800H02	0.01	107	12.7
	0.1	93.0	13.2
M800H07	0.01	106	10.4
	0.1	99.5	9.6
M800H08	0.01	111	12.4
	0.1	98.2	9.0
M800H15	0.01	91.6	17.6
	0.1	83.5	10.0
M800H22	0.01	92.8	22.1
	0.1	103	11.9